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WSC – CAM – III A

Quality Assurance and Quality Control Requirements and Performance Standards for *SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)* for the Massachusetts Contingency Plan (MCP)

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III   Trace Metal Analyses

A.   QA/QC Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by ICP-AES

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1.0 QA/QC Requirements for SW-846 Method 6010B

1.1 Method Overview

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable for all of the analytes listed in Section 2.4 as well as numerous other elements (refer to Table 1, SW-846 Method 6010B). All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been pre-filtered and acidified do not require acid digestion. Samples that are not digested must either use an internal standard or be matrix matched with the standards. Refer to Chapter 3.0, SW-846 Method 6010B for the appropriate digestion procedures.

1.1.1 Reporting Limits for SW-846 Method 6010B

Reporting Limits (RL), sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1, SW-846 Method 6010B lists the recommended analytical wavelengths and estimated instrumental detection limits (IDLs) for numerous elements, including all analytes listed in Section 2.4, in clean aqueous matrices. Elements and matrices other than those listed in Table 1, SW-846 Method 6010B may be analyzed by this method if performance at the concentration ranges of interest (see Section 8.0, SW-846 Method 6010B) is demonstrated.

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for trace metals analyzed in support of MCP decision-making are presented in Appendix III A–1 of this document and Appendix VII-A, WSC-CAM-VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

1.1.2 General Quality Control Requirements of SW-846 Method 6010B

Each laboratory that uses SW-846 Method 6010B is required to operate a formal quality assurance program to demonstrate the precision and bias of the method as performed by the laboratory and procedures for determining the method reporting limit (RL). The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks as a test of continued performance, and the analysis of laboratory control samples (LCSs), and LCS duplicates to assess accuracy and/or precision. Project-specific matrix duplicates or matrix spike duplicates (MSDs) may be used in lieu of LCS duplicates to evaluate precision when such samples are analyzed either at discretion of laboratory or at request of data-user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must

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meet or exceed the performance standards as presented in Section 1.4 and Table III A-1 of this method. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Chapter One, Section 4.4.1 and SW-846 Method 6010B, Section 8.0. The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data-users on request. The data associated with the Initial Demonstration of Proficiency for SW-846 Method 6010B must include the following:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-III A, Table III A-1
Continuing Calibration	WSC-CAM-III A, Table III A-1
Method Blanks	WSC-CAM-III A, Table III A-1
% Percent Recovery for MS/LCS	WSC-CAM-III A, Table III A-1
Relative Percent Difference (RPD) for MSD/LCS Duplicate	WSC-CAM-III A, Table III A-1
Other Instrument QC Samples	WSC-CAM-III A, Table III A-1

It is essential that laboratory-specific performance criteria for LCS, LCS duplicates (or project-specific matrix duplicates or matrix spike duplicates, see Table III A-1) and the other data quality indicators, listed in Table III A-1, also be calculated and documented. When experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices, the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent quality control performance standards described in Table III A-1 to assess analytical trends (i.e., systematic bias, etc) and to improve overall method performance.

For SW-846 Method 6010B, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed on Table III A-1. It should be noted that the performance standards listed in Table III A-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory. Laboratories are encouraged to continually strive to minimize variability and improve the accuracy and precision of their analytical results. The data user will utilize this analytical performance data to verify that the results reported by the laboratory are consistent with the pre-established data quality objectives for the MCP disposal site.

This method is restricted to use by, or under the supervision of spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

1.2 Summary of Method

Solubilized or digested samples are nebulized and the resulting aerosol is transported to the plasma torch using an argon gas carrier. The resulting multi-element emissions produced by a radio-frequency inductively coupled plasma are resolved into its component radiation (wavelengths) by a grating spectrometer. The light emitted by the atoms or ions in the ICP is

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converted to electrical signals by the photomultiplier in the spectrometer. The intensity of the electron signal is compared to previous measured intensities of known concentration of the element and a concentration is computed.

Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line.

For environmental applications, an ICP-AES typically incorporates sequential or simultaneous optical systems and axial or radial viewing of the plasma for multi-elemental determinations.

1.2.1 Sample Preparation

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods as described in Chapter 3.0, SW-846 Method 6010B and summarized in Appendix III A-1, Sample Preservation, Container and Analytical Holding Time Specifications for Surface Water, Groundwater, Soil and Sediment Samples Analyzed for Metals by ICP-AES in Support of MCP Decision Making. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

1.3 Method Interferences

Types of interference and approaches for minimizing their adverse effects are described in detail in Section 3.0, SW-846 Method 6010B, and summarized herein. Tests for the evaluation of potential method interferences are also described in Section 8.5, SW-846 Method 6010B. Types of interferences on ICP include:

➢ Spectral interferences (described in Section 3.1 of SW-846 Method 6010B) – caused by background emission, stray light from high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. Common spectral interferants, which cause suppression or enhancement of other analytes present in a sample, include aluminum, calcium, iron, and magnesium (though other analytes can also contribute to spectral interference and should be monitored – see Table 2 of SW-846 Method 6010B for a list of 21 potential interelement interferents and the analytes that they affect). Spectral interferences are minimized by using background corrections and interelement corrections, which can be applied either automatically by the ICP data system or manually by the spectroscopist. It is strongly recommended that automatic (computerized) corrections for both background and interelement interferences be utilized during analysis of all MCP samples. If not, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results. In all cases, the acceptable analysis of interference check samples (ICSA and ICSAB, see Table III A-1 for acceptance criteria) provides evidence of acceptable background and interelement corrections.

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- ➢ Physical interferences (described in Section 3.2 of SW-846 Method 6010B) – caused by sample viscosity and surface tension affection the sample nebulization. Samples with high dissolved solids or high acid content can exhibit physical interference. Physical interferences can be minimized by diluting the sample, using an internal standard, or using a high solids nebulizer to introduce the sample to the ICP. The common use of mass flow controllers also minimizes the effects of physical interferences and improves ICP performance.

- ➢ Chemical interferences (described in Section 3.3 of SW-846 Method 6010B) – molecular compound formation, ionization effects, and solute vaporization. Chemical interferences are uncommon in the analysis of samples by ICP.

- ➢ Memory interferences (described in Section 3.4 of SW-846 Method 6010B)- caused by a high concentration sample contributing to signal of subsequent sample. Optimizing rinse times between sample analyses (including both field and QC samples) will minimize the potential for memory interferences.

- ➢ High salt concentrations (described in Section 3.5 of SW-846 Method 6010B) – cause analyte signal suppression (e.g., seawater samples). Samples with high salt content can cause both physical interference, by salting-over the torch, and significant suppression of analyte response in the sample. Samples should be diluted to bring the sodium (and other analytes) within the linear range of the instrument; note, however, this approach may raise the sample-specific reporting limit for analytes of interest above the MCP requirements. Therefore, it is recommended that alternate preparation/extraction methods be used to remove the salt interference prior to ICP analysis.

1.4 Quality Control Requirements for SW-846 Method 6010B

1.4.1 General Quality Control Requirements for Determinative Inorganic Methods

Refer to SW-846 Chapter One for general quality control procedures for all inorganic methods, including SW-846 Method 6010B. These requirements ensure that each laboratory maintain a formal quality assurance program and records to document the quality of all inorganic data.

Quality Control procedures necessary to evaluate the instrument's operation may be found in Chapter One, Section 2.0, and include evaluation of calibrations and performance of sample analyses.

Instrument quality control and method performance requirements for the ICP-AES system may be found in SW-846 Method 6010B, Sections 8.0 and 9.0, respectively.

1.4.2 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B

Specific QA/QC requirements and performance standards for SW-846 Method 6010B are presented in Table III A-1. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide a data user with "Presumptive Certainty" regarding the usability of analytical data to

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support MCP decisions. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty", parties must:

(a) Comply with the procedures described and referenced in WSC-CAM–III A;

(b) Comply with the applicable QC analytical requirements prescribed in Table III A-1 for this test procedure;

(c) Evaluate, and narrate, as necessary, compliance with performance standards prescribed in Table III A-1 for this test method; and

(d) Adopt the reporting formats and elements specified in the CAM

In achieving the status of "Presumptive Certainty", parties will be assured that analytical data sets:

✓ Will satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data;

✓ May be used in a data usability assessment, and if in compliance with all MCP Analytical Method standards, laboratory QC requirements, and field QC recommended limits and action levels, the data set will be considered useable data to support site characterization decisions made pursuant to the MCP; and

✓ May be used to help support a data representativeness assessment.

Widespread adherence to the "Presumptive Certainty" approach will promote inter-laboratory consistency and provide the regulated community with a greater degree of certainty regarding the quality of data used for MCP decision-making. The issuance of these requirements and standards is in no way intended to preempt the exercise of professional judgement by the LSP in the selection of alternative analytical methods. However, parties who elect not to utilize the "Presumptive Certainty" option have an obligation, pursuant to 310 CMR 40.0017 and 40.0191(2)(c), to demonstrate and document an overall level of (laboratory and field) QA/QC, data usability, and data representativeness that is adequate for and consistent with the intended use of the data.

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Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency - Daily following profiling and optimization of ICP and prior to sample analysis (2) Minimum of a calibration blank plus one calibration standard for each analyte (may be mixed-analyte solutions). (3) Linear curve fit with correlation coefficient r > 0.995. Second order curve fit may be used if r > 0.995.	No	Re-optimize instrument set up; re-calibrate as required by method.	Linear curve criteria applicable to calibration curves with one blank and 2 or more calibration standards.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency - Daily immediately after initial calibration (2) Separate-source from calibration standards (3) ICV % recovery must be 90-110%; RSD < 5% for the replicate (minimum of 2) integrations.	No	Re-calibrate/Re-analyze ICV as required by method	Suspend all analyses until Initial Calibration non-conformance is rectified.
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift and contamination evaluation)	(1) Frequency - Daily immediately after ICV (2) Must be matrix-matched (the same concentration of acids as standards and samples) (3) ICB must be < Reporting Limit (RL)	No	Re-calibrate/Re-analyze ICB as required by method	
Low Level Calibration Check Standard	Laboratory Analytical Sensitivity (to support the RL)	Only required if NOT including the RL as the low-level standard in the calibration curve. (1) Frequency - Daily prior to field sample analysis (2) Check Standard at the level of the RL for all analytes (3) % recovery recommended 70-130%	No	Re-calibrate / narrate	This is a MADEP-MCP required performance standard. However, it is not required if the RL is supported by a low-level standard in the initial calibration. Narrate non-compliance.
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency - Every 10 samples and at end of run (2) Same-source as calibration standards; near mid-point of linear range (3) CCV % recovery must be 90-110%; RSD < 5% for the replicate (minimum of 2) integrations.	No	Re-calibrate/Re-analyze all samples since last compliant CCV.	Narrate non-compliance.

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Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift and contamination evaluation)	(1) Frequency - Every 10 samples and at end of run immediately after CCV (2) Must be matrix-matched (the same concentration of acids as standards and samples) (3) CCB must be < RL	No	Re-calibrate/Re-analyze all samples since last compliant CCB.	Narrate non-compliance.
Interference Check Standards (ICS A and ICSAB)	Laboratory Analytical Accuracy	(1) Frequency - Daily prior to field sample analysis and at end of run. (2) ICS A and ICSAB contain known amounts of interferants and analytes (see method) (3) ICSA/AB % recoveries for all analytes must be 80-120%	No	May require adjustment of interference corrections, background corrections, and/or linear ranges.	Narrate non-compliance.
Method (Preparation) Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency - One per digestion batch of < 20 field samples. If samples undigested (dissolved metals) the ICB = method blank. (2) Must be matrix-matched (the same concentration of acids as calibration and QC standards) and digested with the samples (3) Method Blank must be < RL	Yes	Re-digest/Re-analyze all associated samples unless all detected results are > 10x method blank level.	Narrate non-compliance.
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	(1) Frequency - One per digestion batch of < 20 field samples. If samples are undigested (dissolved metals) an ICV = LCS. (2) LCS must be matrix-matched to samples (aqueous/solid) and digested with the samples (3) LCS % recoveries for all analytes must be 80-120% for aqueous media and vendor control limits (95% confidence limits) for solids.	Yes	Re-digest/Re-analyze all associated samples.	Narrate non-compliance.

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Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
LCS Duplicate	Laboratory Method Precision	(1) Frequency - One per digestion batch of ≤ 20 field samples. If samples are undigested (dissolved metals) an ICV duplicate may be substituted for an LCS duplicate. (2) Prepared using same standard source and concentration as LCS. (3) Recommended to be run immediately after LCS in analytical sequence. (4) LCS duplicate must be matrix-matched to samples (aqueous/solid) and digested with the samples (5) Laboratory–determined Relative Percent Difference (RPD) must be ≤ 20 (aqueous) and ≤ 30 (solids), and (6) A project-specific MD or MSD may be substituted to evaluate precision in lieu of an LCS duplicate.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Narrate non-conformances
Project-specific Matrix Spike Sample (MS)	Method Accuracy in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples if requested by data-user (see Appendix III A-2 for required matrix frequency) . If samples are undigested (dissolved metals) perform an analytical spike. (2) MS % recoveries for all analytes 75-125% for all media (recommended).	Yes Only when requested by the data-user	If MS % recovery is > 30%, and LCS was in-control, no corrective action is required. If MS %recovery is < 30%, and non-detected results were found, one or more of the following may be performed to confirm matrix effect: 1) homogenize sample well, re-digest, and re-analyze sample/MS; 2) perform method of standard additions for quantitation; 3) perform serial dilution; 4) perform post-digestion spike.	Narrate non-compliance. Note to data users: USEPA Region I data validation guidance requires rejection of non-detected results with MS <30% recovery. Therefore, the 1st corrective action listed may be required for critical sample data to obtain usable results.

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Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Project-specific Matrix Duplicate Sample (MD) or Matrix Spike Duplicate (MSD)	Method Precision in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples, at discretion of laboratory or at request of data-user .If samples are undigested (dissolved metals) perform an analytical duplicate. (2) MD: Prepared by digesting and analyzing an additional aliquot of a field sample. (3) MSD: Prepared by fortifying an additional aliquot of the field sample used for MS. (4) MD relative percent difference (RPD): aqueous results > 5x RL: $\pm$ 20%; aqueous results < 5x RL difference $\leq$ RL; solid results > 5x RL: $\pm$ 35%; solid results < 5x RL : difference $\leq$ 2x RL.	Yes Only when requested by the data-user	Check LCS; if recoveries acceptable in LCS, narrate nonconformance.	Note exceedances in Environmental Laboratory case narrative.
Linear Range Analysis	Laboratory Analytical Accuracy	(1) Frequency - Annually for most metals, every 6 months for those metals that periodically approach the upper limit. (2) Determine the upper limit of the linear dynamic range for each wavelength utilized by determining the signal responses from a minimum of 3 (preferably 5) different concentration standards across the range, see method for details.	No	Not applicable.	See method for requirements. Data must be kept on file to document initial demonstration of performance (IDP) of linear range analysis for each ICP instrument.
Inter-element Spectral Interference Correction Analysis	Laboratory Analytical Accuracy	(1) Frequency – Inter-element spectral corrections must be verified every 6 months. (2) Routine analysis of ICSA and ICSAB verifies inter-element spectral interference corrections. (3) See method for details.	No	Not applicable.	See method for requirements. Data must be kept on file to document initial demonstration of performance (IDP) of interelement correction factors/equations and background correction points for each ICP instrument.

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Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting	NA	(1) Non detected values must be reported with the sample-specific reporting limit for each ICP analyte. (2) The RL must be $\leq$ the applicable regulatory compliance standard for each metal reported (3) The RL must be verified at least daily with a low-level calibration check standard following the calibration curve or supported by the low-level standard in the calibration curve. (4) Results for soils/sediments must be reported on a dry weight basis for comparison to MCP regulatory standards (5) Sample concentrations that exceed the highest calibration standard must be diluted (in the same acid matrix) to fall within the calibration range when reanalyzed	Yes	Not applicable.	

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1.5 List of Analytes for SW-846 Method 6010B

The MCP analyte list for SW-846 Method 6010B presented in Table III A-2, is intended to be protective of human health and the environment and the Commonwealth's groundwater resources. The list is comprised of potential toxic metal contaminants that are readily-analyzable by SW-846 Method 6010B and have a Method 1 Groundwater/Soil Standards as described in 310 CMR 40.0974

The MCP Method 1 Groundwater/Soil Standards used to characterize the risk of harm posed by oil or hazardous materials at a disposal site are described in 310 CMR 40.0974(2), Table 1. This list of groundwater/soil standards, developed by the Department, takes into account a defined set of conservative potential exposure pathways likely to be encountered at most disposal sites. Method 1 Standards have been developed by the Department for over one hundred organic and inorganic contaminants that are commonly encountered at MCP disposal sites.   The MCP Method 1 Groundwater/Soil Standards list is periodically reviewed and updated by the Department. When compounds are added to the MCP Method 1 Groundwater/Soil Standards list that are suitable for analysis by SW-846 Method 6010B, the analyte list for this method will be updated accordingly.

1.6 Additional Analyte Reporting Requirements for SW-846 Method 6010B

1.6.1 Analysis and Reporting recommendations for SW-846 Method 6010B

While it is not necessary to request and report all the SW-846 Method 6010B analytes listed in Table III A-2 to obtain Presumptive Certainty, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Uncharacterized sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

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✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note that a desire to avoid detection and quantitation of a contaminant that is present or likely present at a site above background levels is not a valid reason to limit an analyte list, and that such an action could constitute a criminal violation of MGL c. 21E.

In cases where a truncated list of method analytes is selected, laboratories must still employ the method-specific quality control requirements and performance standards associated with the requested analytes list to obtain Presumptive Certainty status.

1.6.2 Elements Used to Evaluate Inter-Element Spectral Interferences

Elements not listed in Table III A-2 and identified and quantified in the course of SW-846 Method 6010B analysis of field samples to evaluate inter-element spectral interferences, etc., need not be reported as contaminants.

1.6.3 Special Analytical and Reporting Considerations for Mercury

Because of its elevated Estimated Instrumental Detection Limit (Table 1, SW-846 Method 6010B) Mercury, a common environmental contaminant, is not included on Table III A-2, Analyte List for SW-846 Method 6010B.

Although Mercury is not required to be reported to obtain "Presumptive Certainty" status for SW-846 Method 6010B, it must be given consideration as a contaminant of concern when sites with unknown, uncertain or complex history are assessed for potential contamination associated with "total metals" to satisfy due diligence requirements pursuant to 310 CMR 40.019. Under these circumstances, the preferred analytical method for Mercury is SW-846 Method 7470/7471 (Cold Vapor AAS).

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Table III A-2 Analyte List for SW-846 Method 6010B

ANALYTE	CASRN	MCP METHOD 1	
		GW-1	S-1/GW-1
		µG/L (PPB)	mG/KG (PPM)
Antimony	7440360	6[1]	10
Arsenic	7440382	50	30
Barium	7440393	2000	1000
Beryllium	7440417	4	0.7
Cadmium	7440439	5	30
Chromium (Total)	7440473	100	200[2]
Lead	7439921	15[1]	300
Nickel	7440020	100	300
Selenium	7782492	50[1]	400
Silver	7440224	7 (GW-3) 40 (GW-1)	100
Thallium	7440280	2[1]	8
Vanadium	7440622	50	400
Zinc	7440666	900 (GW-3) 2000 (GW-1)	2500

1. Laboratory Reporting Limits for this element may not be able to achieve MCP regulatory compliance limit using SW-846 Method 6010B.
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2.0 Data Usability Assessment for Method 6010B

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 Method 6010B which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document.

3.0 Reporting Requirements for SW-846 Method 6010B

3.1 General Reporting Requirements for SW-846 Method 6010B

General reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VIIA. This guidance document provides recommendations for field QC, as well as the required content of the Environmental Laboratory Report, including

- ➢ Laboratory identification information presented in WSC-CAM-VII A, Section 2.4.1,
- ➢ Analytical results and supporting information in WSC-CAM-VII A, Section 2.4.2,
- ➢ Sample- and batch-specific QC information in WSC-CAM-VII A, Section 2.4.3,
- ➢ Laboratory Report Certification Statement in WSC-CAM-VII A, Section 2.4.4,
- ➢ Copy of the Analytical Report Certification Form in WSC-CAM-VII A, Exhibit VII A-1,
- ➢ Environmental Laboratory case narrative contents in WSC-CAM-VII A, Section 2.4.5,
- ➢ Chain of Custody Form requirements in WSC-CAM-VII A, Section 2.4.6

3.2 Specific Reporting Requirements for SW-846 Method 6010B

Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B are presented in Table III A-1. Specific reporting requirements for SW-846 Method 6010B are summarized below in Table III A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis.

3.2.1 Reporting Data for Spectral Interference Corrections

If unusual inter-element or matrix interferences are encountered, a description of any corrective measures utilized by the laboratory must be included in the Environmental Laboratory case narrative. Such measures may include use of an alternative analytical wavelength, non-standard computerized compensation, sample dilution to overcome physical or chemical interferences, use of standard additions, or other method-specific corrective actions.

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Table III A-3 Routine QA/QC Reporting Requirements for SW-846 Method 6010B

Parameter	Required Analytical Deliverable
Initial Calibration	*NO*
Initial Calibration Verification (ICV)	*NO*
Initial Calibration Blank (ICB)	*NO*
Low Level Calibration Check standard	*NO*
Continuing Calibration Verification (CCV)	*NO*
Continuing Calibration Blank (CCB)	*NO*
Interference Check Standards (ICS A and B)	*NO*
Method (Preparation) Blank	*YES*
Laboratory Control Samples (LCS)	*YES*
LCS Duplicate (or project-specific MD or MSD)	*YES*
Project-specific Matrix Spike Sample (MS)	*YES, only if requested by the LSP*
Project-specific Matrix Duplicate (MD)	*YES, only if requested by the LSP*
Project-specific Matrix Spike Duplicate (MSD)	*YES, only if requested by the LSP*
Linear Range Analysis	*NO*
Inter-element Spectral Interference Correction Analysis	*NO*
General Reporting – sample specific reporting limits	*YES*

Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

4.0 Regulatory Limits for Metals under 310 CMR 40.000

The most stringent (lowest) MCP Reportable Concentrations (RCs) and Method 1 Standards for metals analyzable by SW-846 Method 6010B are as follows:

Metal	RQ Pounds	RC GW-1 mg/L - (ppm)	RC S-1 mg/kg - (ppm)	Method 1 Groundwater ug/L - ppb	Method 1 Soils ug/g - ppm
Antimony	50	0.006	10	6	10
Arsenic	1	0.05	30	50	30
Barium	100	2	1000	2000	1000
Beryllium	5	0.004	0.7	4	0.7
Cadmium	5	0.005	30	5	30
Chromium (III)	100	0.1	1000	100	1000
Chromium (VI)	100	0.1	1000	50	200
Cobalt	50	5	500	NS	NS
Copper	100	10	1000	NS	NS
Lead	5	0.02	300	15	300
Lithium	10	1	100	NS	NS
Mercury[1]	1	0.001	20	2	20
Nickel	10	0.08	300	80 (GW-3)	300
Phosphorous	1	NA	NA	NS	NS
Potassium	10	NA	NA	NS	NS
Selenium	10	0.05	400	50	400
Silver	50	0.007	100	7 (GW-3)	100
Sodium	5	NA	NA	NS	NS
Thallium	50	0.002	8	2	8
Vanadium	50	0.05	400	50	400
Zinc	50	0.9	2500	900 (GW-3)	2500

NA – Not Applicable
RQ – Reportable Quantity
RC – Reportable Concentration for Groundwater (GW-1) and Soils (S-1)
Method 1 Groundwater – GW-1 Category unless otherwise noted
Method 1 Soils – Category S-1/GW-1 in all cases
NS – No MCP Method 1 Standard has been promulgated by the Department.
1 Mercury values presented for completeness only. Analyze mercury by
   SW-846 Methods 7470A and 7471

Title: Sample Collection, Preservation, And Handling Procedures for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, sediment and wastes matrices for metal analyses conducted in support of MCP decision-making are summarized below and presented in Appendix VII-A of WSC-CAM-VIIA, Quality Assurance and Quality Control Guidelines for Sampling, Data Evaluation, and Reporting Activities for the Massachusetts Contingency Plan (MCP). Additional guidance may be found in SW-846, Chapter Three

Matrix	Sample Container(s) [1]	Preservative	Holding Time [2]
Total Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for Total Metals	HNO_3 to pH < 2,	180 days: all metals except mercury 28 days: mercury
Dissolved Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for field-filtered sample for Dissolved Metals	Filter (0.45 µm) unpreserved sample on site; or at the laboratory (*prior to acid preservation)* within 24 hours of collection, then HNO_3 to pH <2,	180 days: all metals except mercury 28 days: mercury
Suspended Metals Groundwater and Surface Water	Submit Suspended Solids on Filter to Laboratory	Filter on site Filter 100 – 500 ml of unpreserved sample	180 days: all metals except mercury 28 days: mercury
Soils and Sediments	(1) 4-ounce glass jar	Cool, 4°C	180 days: all metals except mercury 28 days: mercury
Concentrated Waste Samples	125 mL wide mouth glass or plastic	Cool to 4°C	180 days: all metals except mercury 28 days: mercury

1 The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised.
2 From date of sample collection.

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Title: Methods for Sample Digestion or Preparation by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

SW-846 Method	Method Description
3005	Method prepares ground water and surface water samples for total recoverable and dissolved metal determinations by FLAA, ICP-AES, or ICP-MS. The unfiltered or filtered sample is heated with dilute HCl and HNO prior to metal determination.
30l0	Method prepares waste samples for total recoverable metal determinations by FLAA, ICPAES, or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid. The method is applicable to aqueous samples, EP and mobility-procedure extracts.
3015	Method prepares aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total recoverable metal determinations by FLAA, GFAA, ICP-AES, or ICP-MS. Nitric acid is added to the sample in a Teflon digestion vessel and heated in a microwave unit prior to metals determination.
3031	Method prepares waste oils, oil sludges, tars, waxes, paints, paint sludges and other viscous petroleum products for analysis by FLAA, GFAA, and ICP-AES. The samples are vigorously digested with nitric acid, sulfuric acid, hydrochloric acid, and potassium permanganate prior to analysis.
3040	Method prepares oily waste samples for determination of soluble metals by FLAA, GFAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content
3050	Method prepares waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples.
3051	Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid is added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination.
3052	Method prepares siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total analysis by FLAA, CVAA, GFAA, ICPAES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis